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Technical Report 32-1179

A Mechanical Tension Testing Facility for Brittle Materials

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Abstract

A mechanical testing facility for measurement of deformation and fracture stresses in tension has been designed, constructed, and evaluated. Special consideration has been given to its use with brittle ceramic materials. The unit makes use of air-floated spherical bearings and close dimensional tolerances along with controlled assembly procedure to reduce axial misalignment. Strains as small as 2×10^{-5} are measured with a pair of commercial optical motion trackers. These trackers avoid introduction of bending moments or mechanical attrition to the specimen surface. Calculation of the magnitude and effect of misalignment are presented. A special streamline contour at the specimen shoulder reduces notch effect. Environmental control permits use of the system in various atmospheres to a maximum temperature of 2500°C.

After construction, the capability of the unit has been evaluated to ascertain limits of accuracy. The limits appear to be as follows:

(1) Average stress $\leq$1% of full scale (load/unit area).

(2) Local stress $\leq$5% of full scale.

(3) Strain sensitivity 2×10^{-5}.

(4) Strain precision 2% of full scale $+ 2 \times 10^{-5}$.

(5) Meaningful modulii with strains of 2.5×10^{-3}.

A Mechanical Tension Testing Facility for Brittle Materials

I. Introduction

Perhaps the most troublesome problem in the present application of ceramic materials is the frequent mechanical failure. This failure is often controlling when the primary function of the ceramic is not of a mechanical nature; as, for example, in the mechanical cracking of the ceramic dielectric in a capacitor. In a program to ascertain the relationship of such mechanical failure to the structure of the material, precise measurements of mechanical behavior are necessary. Initial evaluation of the requirements indicates that modulus of rupture data, which is readily determined, would not be adequate. This inadequacy stems partly from a desire to measure behavior beyond a purely elastic region and partly from the desire to expose a larger volume of material to a uniform stress so that the effect of random structural anomalies in the material might be observed. These anomalies are often considered to be the source of failure in ceramics.

For these reasons, pure tensile testing was selected. This selection does not suggest that tensile testing of brittle materials is without significant problems. Such problems are, for example, the application of pure axial loads and the measurement of the extremely small strains which occur. However, these problems are matters of technique and are not fundamental limitations of the approach. The mechanical testing facility described here has been designed with special emphasis on the testing of brittle materials exhibiting small strains, and the approach was expected to eliminate or minimize the aforementioned problems.

An overall view of the facility, which resulted from these considerations, is shown in Fig. 1. The heart of the facility is a standard mechanical testing frame, to which special air-floated spherical bearings are fixed to improve axiality of loading (Ref. 1). A special dual-range load cell is incorporated to permit wide ranges of load measurement without changes in the alignment. Optical trackers are used to measure the small strains without mechanical attrition on the surface of the specimens or introduction of bending moments from contact with the specimen or grips. A special contour at the specimen shoulder is employed to reduce the notch effect (Ref. 2) in brittle materials. Finally, an environmental chamber is incorporated to be used where the effect of temperature or other environment is considered to be significant, or for protection of associated high temperature components. The consideration for the selection and design of each component and the evaluation of their performance of the entire system will be evaluated.

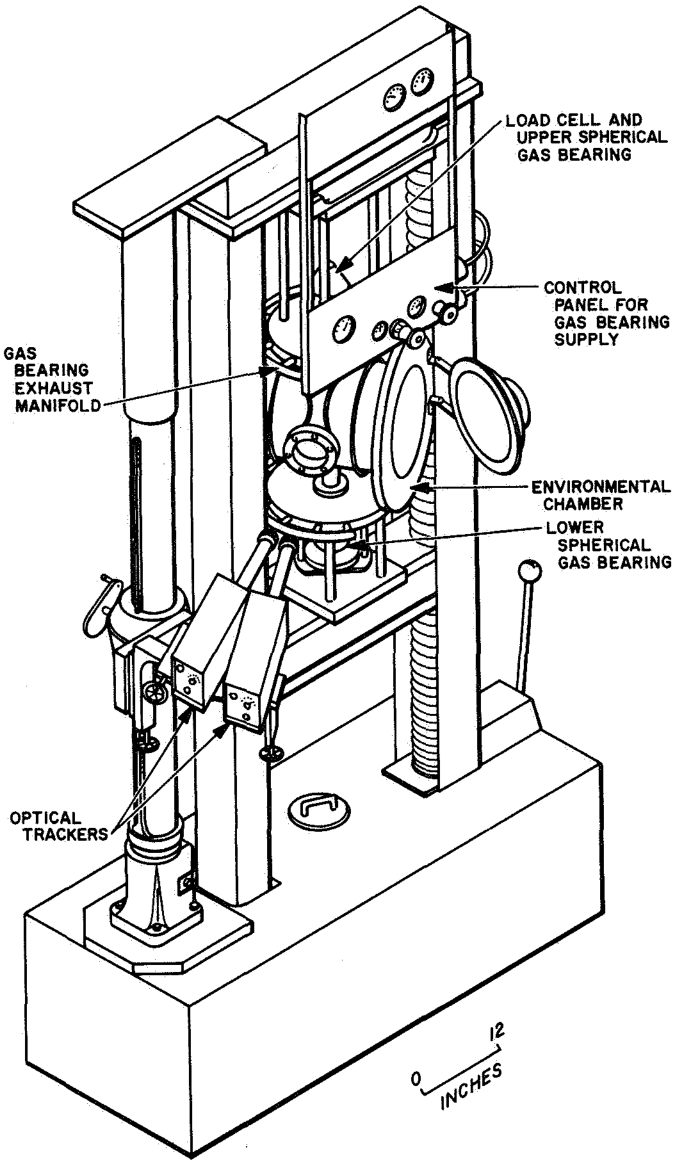


Fig. 1. Mechanical test facility for determination of mechanical properties of brittle materials

II. Frame and Stress System

A. Frame

The frame used in this facility is an extra width, extra length Instron Model TTM. It provides constant rate crosshead motion over a range of 10^{-4} to 0.5 cm/min. Approximately 50% of this range is obtained instantaneously by push-button operation, while the other 50% requires a mechanical gear shift. The drive is servomechanism controlled and provides an accuracy of 0.1% or rated speed over the entire load range.

B. Load Measuring System

The loads are measured by a specially designed and built dual-range load cell. The more sensitive range has a maximum capacity of 100 kg; the less sensitive range is 1000 kg. The cell consists of two cantilever flexures and two unbonded strain gage assemblies (Model 150 Stathern displacement transducers). The mechanical arrangement is such that both ranges read from zero load, and mechanical stops prevent damage to the more sensitive flexure during high load operation. The transducers are mounted to the load cell in a reversed manner; that is, increasing force unloads the transducer. This design was adopted to prevent overloads and mechanical shock from damaging the transducers. Figure 2 shows a plan view of the cell.

The transducers are powered by the standard Instron 390-cycle load cell supply. Individual switching, balance, phase adjust, and calibration controls were incorporated to permit separate operation of either range, and rapid interchange between the two ranges (Fig. 3). Zero suppression and range expansion are available on either range, and a fixed suppression may be preset for use in creep tests. The standard Instron circuitry is used to provide electrical amplification up to 50 times in six scales on either range.

Gage area compensation is also available on either range using the standard Instron system. The final data in terms of load or load per unit area are displayed on a standard strip chart recorder and on an X-Y plot. A 0–1 V dc signal from a follower potentiometer on the strip chart recorder is available for other recording purposes.

Constant load operation may be obtained for creep testing by cycling the crosshead under the control cams. Direct use of this capability is accurate to approximately 1% of full scale load. However, by suppressing the load zero and expanding the load scale, the load may be maintained constant within 1/10 of 1% of full load or better.

Calibration of the load measuring system was made by momentary introduction of fixed resistors across one arm of each transducer bridge. The offset produced was calibrated against a standard load cell connected in place of the load train. The linearity and precision of the low and high ranges were better than ± 0.5 and $\pm 1.0\%$, respectively. The standard cell had been calibrated to an accuracy of $\pm 0.1\%$.

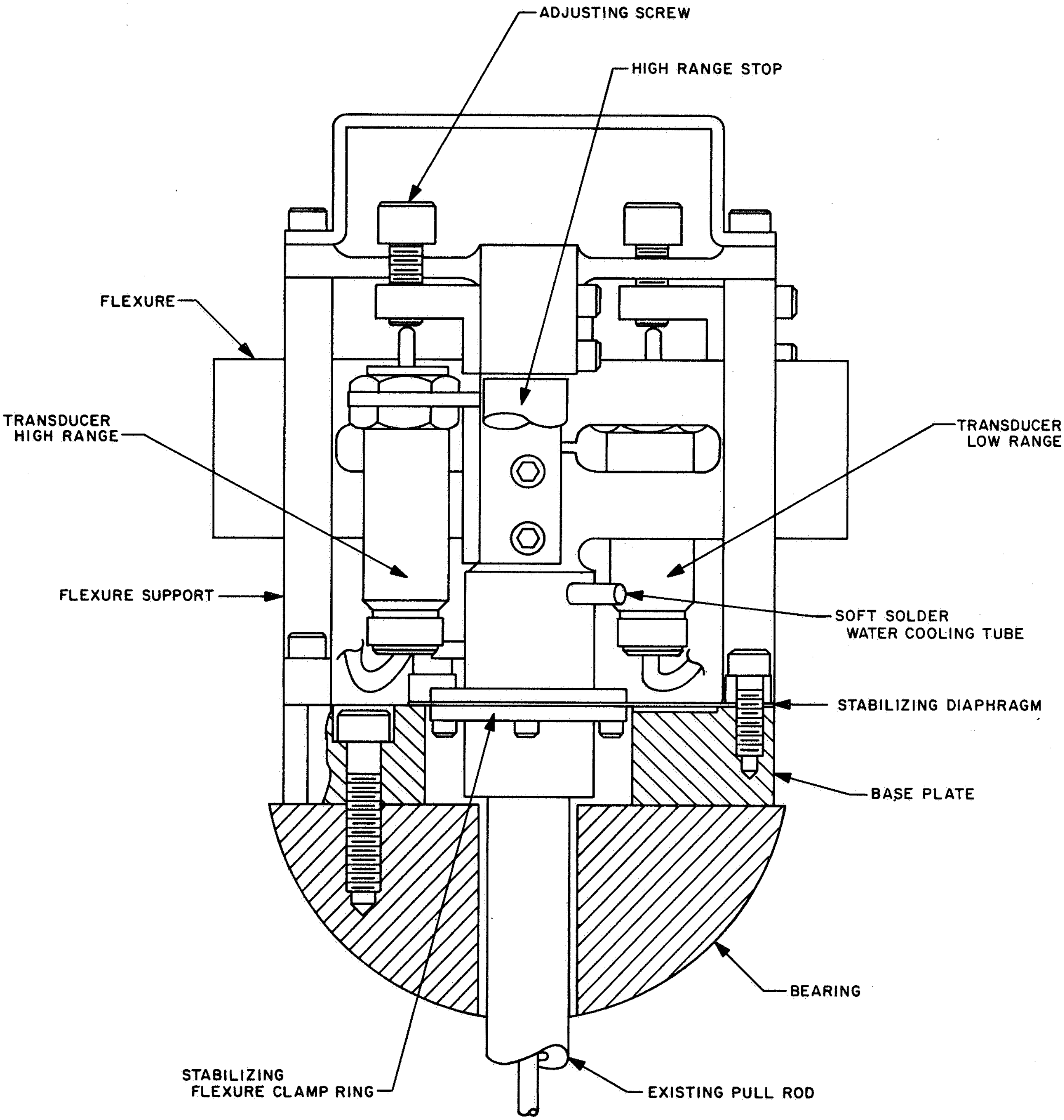


Fig. 2. Dual-range load cell

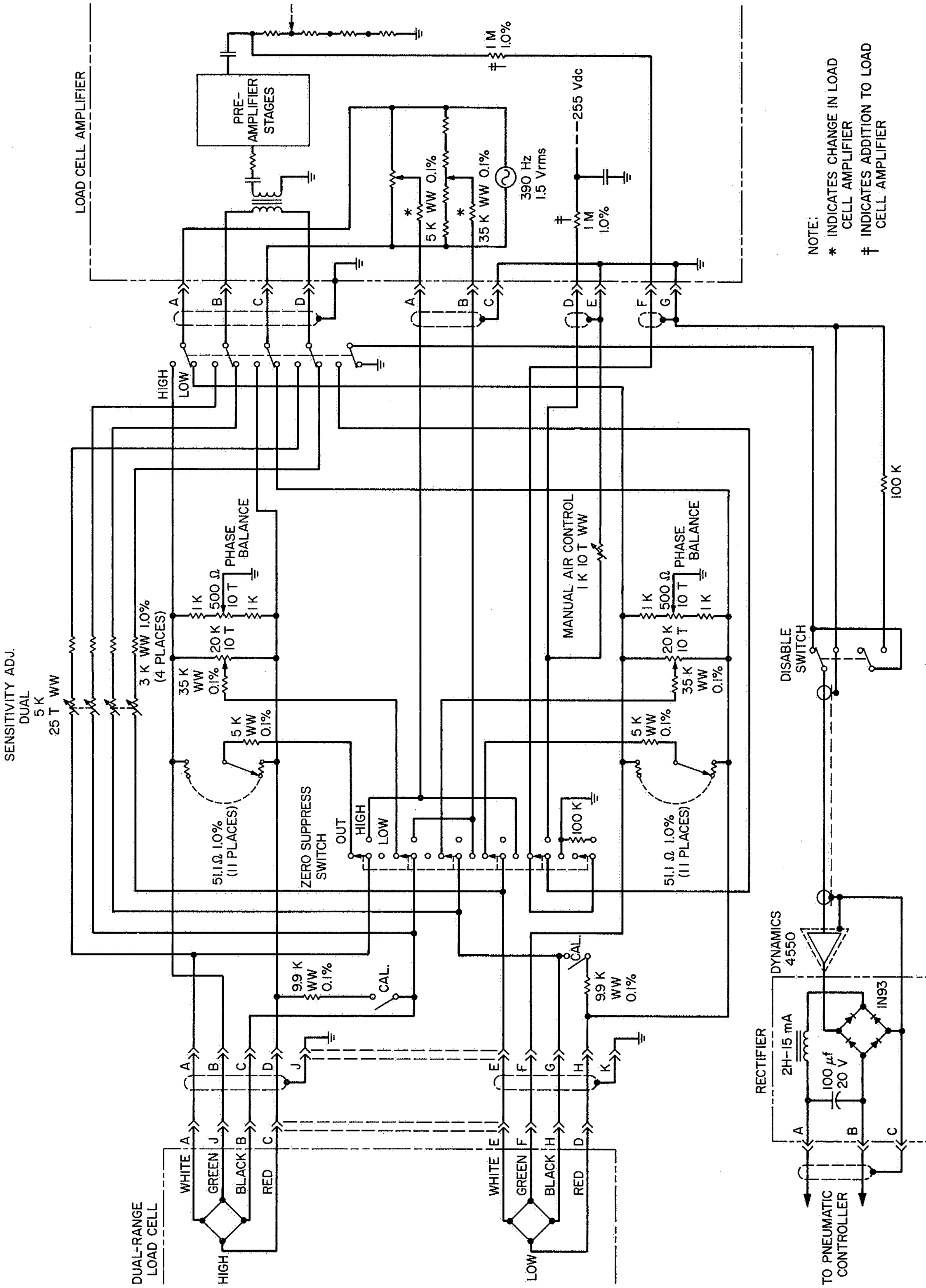


Fig. 3. Schematic diagram for dual-range load train control

III. Strain Detection System

Two optical trackers (Optron Model 650) form the core of the strain detection system. These are electro-optical trackers which follow a light-dark interface and produce a dc output proportional to the position of the interface in the field of view. By using two trackers observing opposite interfaces (opposing specimen shoulders) and adding the electrical outputs, a signal proportional to the strain in the specimen may be obtained. Since linearity of the units is improved when both trackers observe the same relative position of light and dark, one tracker is mounted in an inverted position. By changing lenses, the observable field of view and sensitivity of the trackers may be changed. In this case, two sets of lenses are available; one with a range of 5 mm and a sensitivity of 1μm, and a second with a range of 1.2 mm and a sensitivity of 0.5 μm.

An electrical signal control panel was designed and constructed (Fig. 4) to permit summing and modification of the outputs from the optical trackers. The signals may be attenuated individually to permit calibration and balance of the optical trackers. The signals are then summed and may be electrically filtered at frequencies of 1, 3, 10, 300, 1000, and 3000 Hz. The signal may then be amplified from 0.5 to 20 times and displayed on a strip chart recorder and an X-Y recorder. Outputs are available for high-speed recording equipment.

The optical trackers require a light-dark interface and normally sight on the shoulder of the specimens. At low temperatures, a bright background is required to contrast the specimen; while at high temperatures (>1500°C), specimen self-illumination against a dark background may be used. Since the trackers are sensitive to changes in illumination, both absolute and light-dark ratio, care must be taken in the illumination source. The factors that need to be controlled are: (1) uniformity of intensity with time, and (2) uniformity of intensity across the light source. When operating with backup lights, variations in intensity with time are controlled by using a regulated dc power supply for powering the light. The dc power is required since the trackers are capable of tracking ac flicker. Current regulation to the lamp maintains a light output that is constant within the limits of detection and over the time period of a test. The spacial distribution of light was made uniform by using flat-filament tungsten strip lamps placed such that each tracker sights directly on a filament at all sighting positions. The filaments are aligned by adjusting the positions of the lamps with minimum lens aperture so that the increased depth of field permitted observation of the filament location. The aperture is set to maximum for use, and the light level is adjusted by filters. When operating with self-illumination, it is necessary only to maintain a constant temperature and avoid significant gradients.

Calibration of each optical tracker is accomplished by tracking the motion of the crosshead for a fixed period of time and by adjusting the electronic panel for appropriate response. Next, the two trackers are summed while tracking a specimen moving without load, and the trackers are trimmed to produce zero sum. The normal calibration for the 5-mm lenses is 0.1 strain full scale with unity magnification for specimens with shoulder to

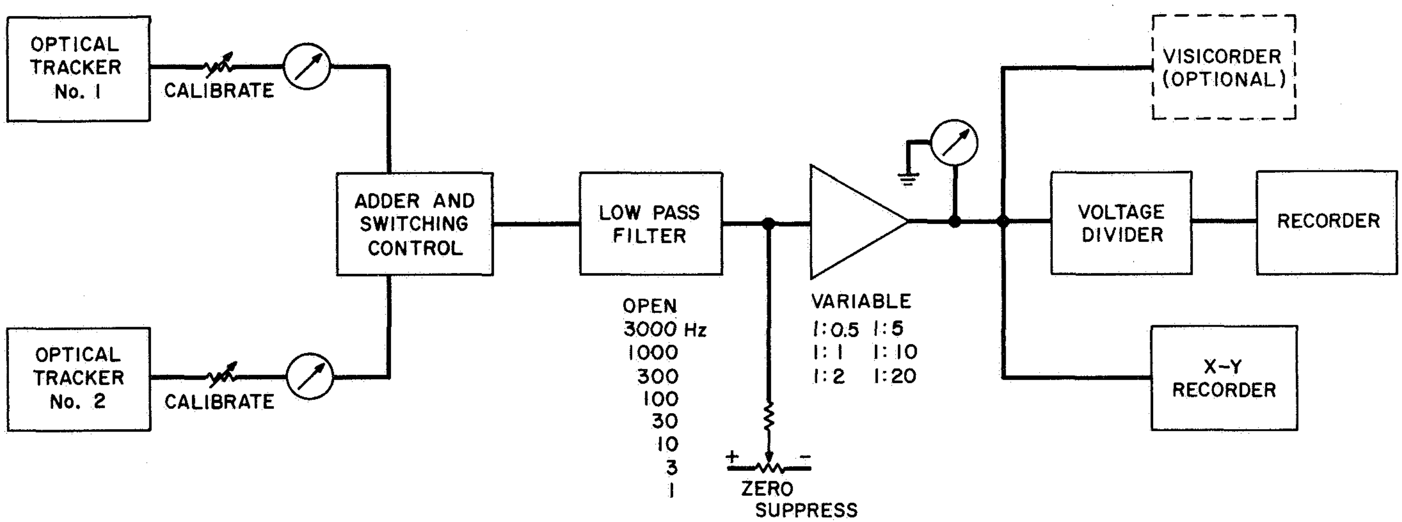


Fig. 4. Electrical schematic for strain detection system

shoulder spacings of 30–40 mm. For electrical magnification, these lenses may be used to sensitivities of 0.00025 strain full scale. The 1-mm range lenses are normally calibrated for 0.025 strain, but the more sensitive ranges down to 0.0000625 are excessively noisy.

It was also necessary to determine the reduction in observed strain from shoulder to shoulder as a result of the increased cross section in the streamline fillet region. This was done with a computer, using the coordinates of the contour and selected constant diameter (gage) lengths. The results are shown in Table 1.

Table 1. Strain correction factor for fillet in streamline contour specimens

Gage length, in.	Ratio of observed strain from shoulder to shoulder to equivalent strain
0.0000	0.8137
0.1250	0.8413
0.2500	0.8617
0.3750	0.8775
0.5000	0.8900
0.6250	0.9003
0.7500	0.9087

IV. Load Train

A. Mechanical Components

The design and the construction of the load train for this mechanical testing facility is probably the most critical area. The concept used was to assemble the entire assembly as a rigid unit. Such assembly was attained by fabrication of the specimen, grip, and the load train elements with such tolerances that the possibility for random misalignment was minimized and to assemble these elements in controlled manner (Fig. 5). This assembly procedure is discussed in Section IV-D. This rigid unit was then placed between two freefloating spheres, such that the specimen load train assembly lay on the line connecting the center of the two balls. This relationship is shown in Fig. 6. The lower assembly is symmetrical with the exception that an additional seat is provided below the lower ball to support the weight of the lower assembly prior to testing.

The pull-rod connectors, shown in Fig. 6, are aligned with the spherical balls to within 0.0001 in. prior to mounting in the frame. These connectors are water-cooled to prevent heating of components beyond this point. The attachment of the pull-rods to these connectors is again by means of close tolerance fit to insure

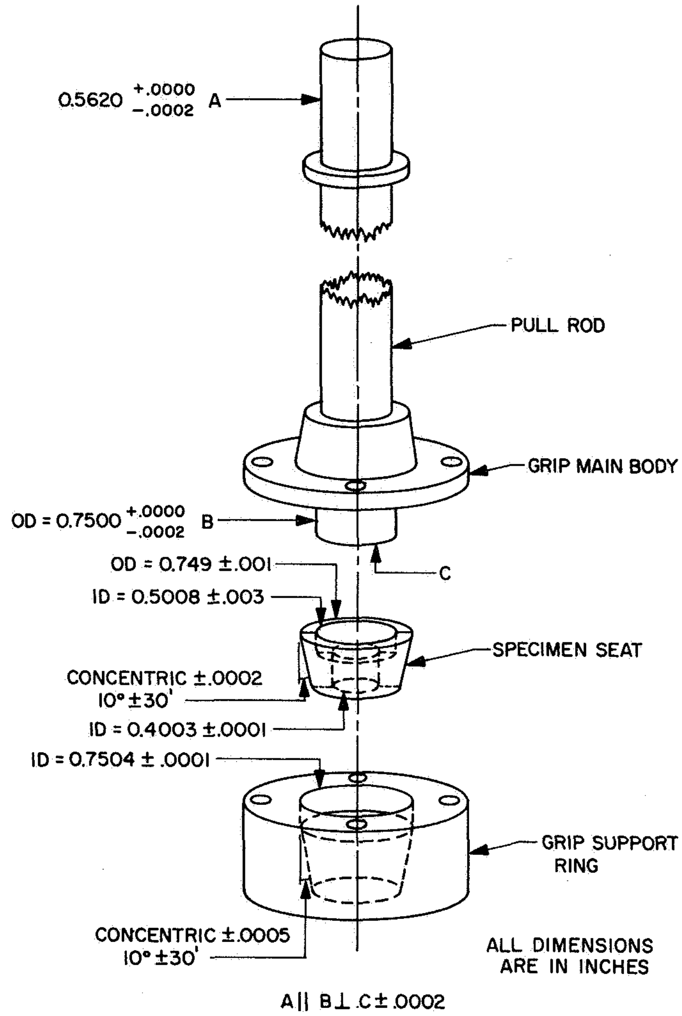


Fig. 5. Specimen grip

that the pull-rod specimen assembly lies on the line connecting the two spherical balls.

B. Gas Bearings

The most suitable technique available for the application of uniform tensile stress appears to be use of gas-floated spherical bearings (Ref. 1). The units employed were 4-in. diam balls requiring a nominal supply pressure of 750 psi to support 1000 kg. The upper bearing is slightly less than a hemisphere, while the lower ball is a full sphere with an additional seat below to support the weight of the ball and load train, thus permitting tests to start from zero stress.

The gas supply for the bearings consist of a bank of gaseous nitrogen cylinders capable of supplying gas to a pressure of 1000 psi. The design of the bearings is such that a gas supply of approximately 5 ft^3/min (measured

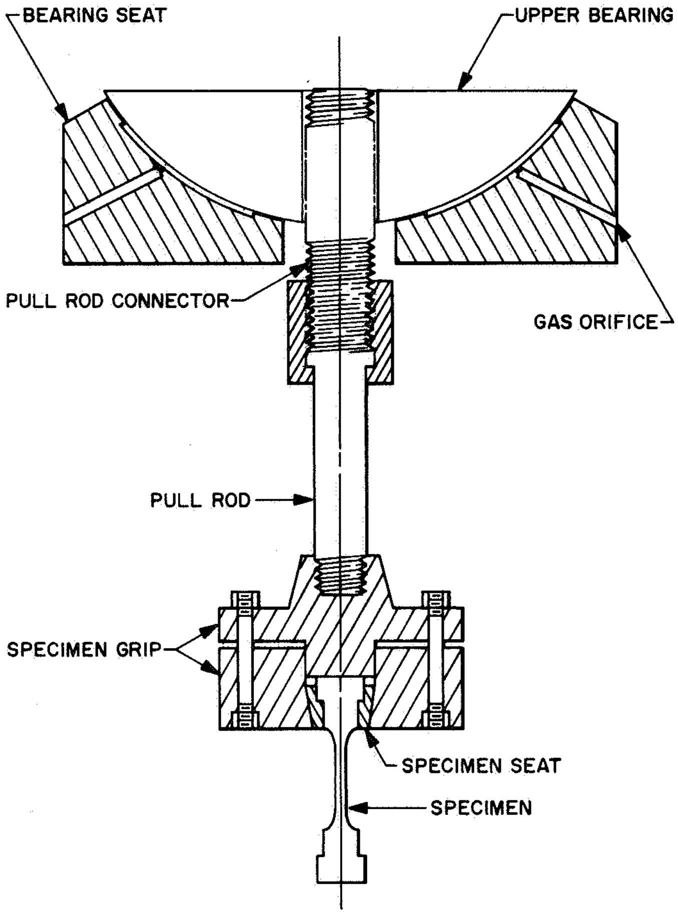


Fig. 6. Load train

at pressure) is required. Since changes in supply pressure to the ball will produce variations in the applied load, it is necessary to control this pressure closely, and especially uniformly, to prevent the application of uncontrolled loads. The supply pressure is regulated toward this end in terms of pressure and volume by the system shown schematically in Fig. 7.

This system for providing control of the supply gas was designed so that the gas pressure above a minimum is proportional to the applied load. This system operates by taking a portion of the load cell signal, amplifying and rectifying it, and using it to drive an electro-pneumatic transducer system (Fisher Governor Co. Model 546 transducer and 530 Gismo regulator). A defeat switch and independent manual control of the air pressure are also provided.

Both bearings are counterbalanced so that, with the complete load train in place except for the specimen, the two seats lie on a vertical line within 0.001 in.

The gas bearings were evaluated only to the extent that a torque of less than 1 in.-oz was sufficient to rotate the entire load train assembly about its own axis to a load of 600 kg. At some point between 600 and 800 kg, resistance to such motion increased. This limitation could have been reduced by increased gas supply pressures, but these higher loads were not used.

C. Specimen Configuration and Evaluation

The specimens for which this facility was designed were produced by hot-pressing (Ref. 3), although other techniques would be equally useful. The blanks used were pressed ¾ in. in diameter by 2½ in. long, and were first ground to 0.55 in. to permit visual inspection for flaws. They were then ground to the form shown in Fig. 8. They were inspected by the procedure listed in the appendix. In some cases, the perpendicularity of the ends did not meet specification. Since this is critical to the proper assembly of the load train, these specimens were salvaged by squaring the ends in a jig with a surface grinder. These parts were then capable of being assembled into the grip assembly with the required precision.

Attempts were made to evaluate the effectiveness of the streamline contour in eliminating notch effects; although the effectiveness of the contour used has apparently been well documented. The technique employed was to apply a coating of Stress Coat ST-80, to stress the specimen, and to study the distribution of stress cracks; however, sensitivity of detection of variation in stress distribution was limited to at best to 10% by the maximum elastic strain which could be introduced into the specimen. At this level, the distribution was uniform; however, uniformity to only 10% would not be satisfactory. Consequently, references to the literature on the streamline contour must be used for higher precision (Ref. 2).

D. Load Train Assembly

Since the fundamental requirement for true axial loading in the design of this system was the assembly of a rigid, straight, and concentric load train, extreme care was required in this operation. Initial trials using strain gaged specimens indicated that, even with careful cleaning and the minimum practical tolerances, uncontrolled assembly did not produce a straight unit. Apparently, invisible amounts of grit or burrs sometimes became lodged between the parts resulting in a non-linear assembly, and subsequent bending moments were detected by the strain gages. This behavior was random with some

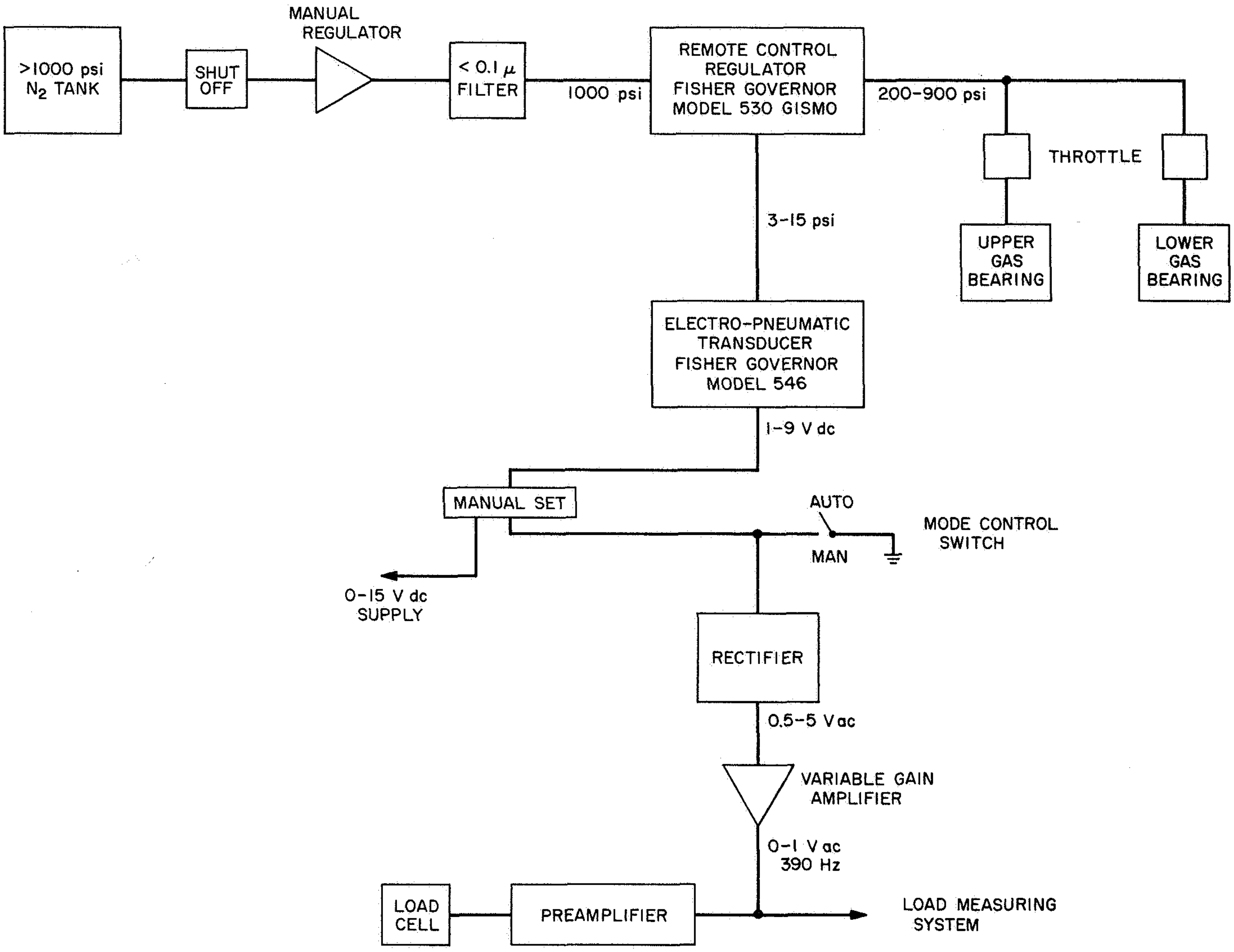
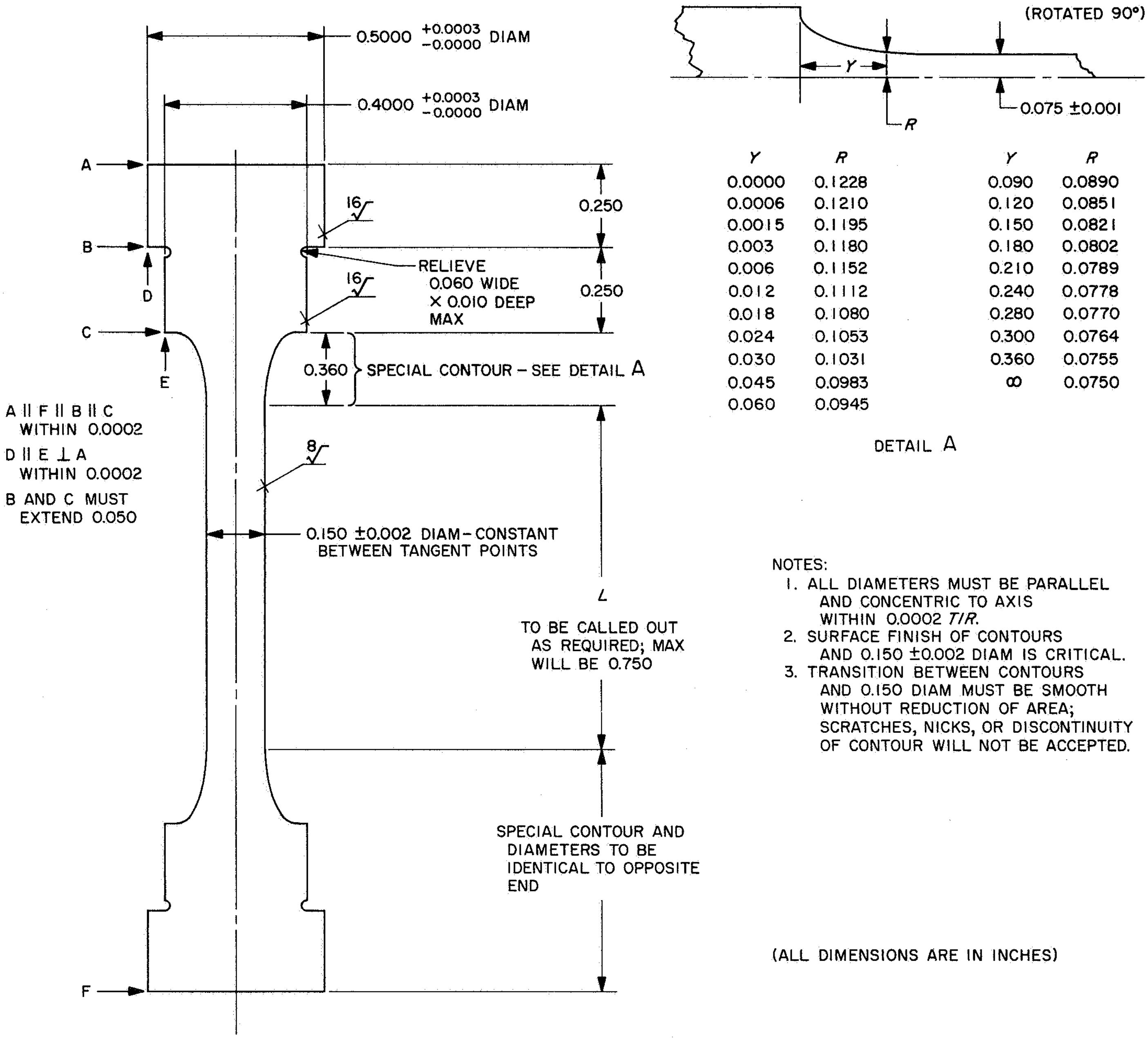


Fig. 7. Schematic of gas bearing gas supply system

assemblies being satisfactory. A variety of assembly jigs consisting of sleeves, V-blocks, etc., were tried but none were sufficient to assure alignment. Consequently, a secondary method of checking the alignment and evaluating alignment without the use of strain gages was required. This method was developed in the form of a 0.0001-in. dial indicator and a pair of differential distance detectors (Bently Scientific Co., Berkeley, Calif.). The upper pull-rod is held in a vertical position in a dummy upper collet, and the specimen and upper grip are assembled. Either the dial indicator or the distance detector may be used to check alignment of the lower end of the specimen within 0.0002 in., while the upper grip is tightened. The other pull-rod is then placed in the dummy collet and the remaining grip is assembled on the specimen. The distance detector is used to check alignment within 0.0005 in. on the lower end of the previously assembled grip pull-rod, while the second grip is tightened. The distance detector, rather than the dial indicator, is required for this second operation, because the side loads introduced by the dial indicator cause bending in the specimen resulting in inaccurate readings.

The ability of this load train system to apply axial stresses to the tensile specimen was determined by means of strain gages. A standard specimen was instrumented by attaching strain gages to the section at 90-deg intervals around the circumference of the specimen. It was found in the course of this evaluation that it was necessary to use a nonplastic material, such as aluminum oxide for the evaluation specimen; since metal evaluation specimens tended to be deformed when nonaxial loads were



Y	R	Y	R
0.0000	0.1228	0.090	0.0890
0.0006	0.1210	0.120	0.0851
0.0015	0.1195	0.150	0.0821
0.003	0.1180	0.180	0.0802
0.006	0.1152	0.210	0.0789
0.012	0.1112	0.240	0.0778
0.018	0.1080	0.280	0.0770
0.024	0.1053	0.300	0.0764
0.030	0.1031	0.360	0.0755
0.045	0.0983	∞	0.0750
0.060	0.0945		

Fig. 8. Tensile specimen

applied. With clearances shown in Fig. 2 and the alignment procedure described, the axiality of loading was approximately 1%, the precision of the strain gages.

E. Effect of Misalignment

Calculations were made to show the effect of misalignment on the stress distribution in a specimen tested in *uniaxial* tension. The geometry of a specimen diameter d being pulled by force F with a misalignment θ is shown in Fig. 9. It is assumed that θ (rad) $<<1$; hence, $\theta = \tan\theta$ and $\cos\theta = 1$. The tensile force on the specimen is

$$\sigma_T = \frac{4F}{d^2}$$

The force due to bending is

$$\sigma_b = \frac{Md}{2I}$$

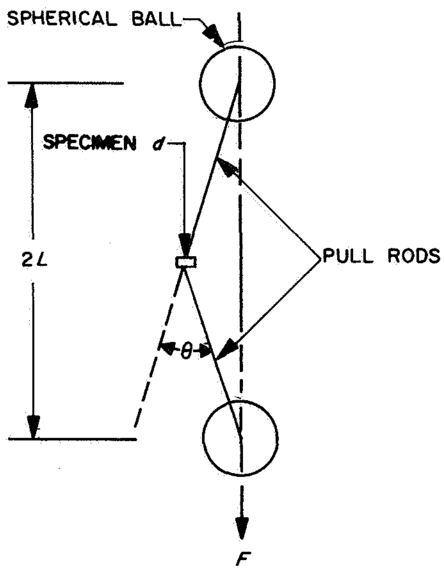


Fig. 9. Representation of misaligned specimen

where M = bending moment $FL\theta$ and I = moment of inertia of specimen of circular cross section $= d^4/64$

The fraction bending is then

$$\frac{\sigma_b}{\sigma_T} = \frac{8\theta L}{d}$$

If the load train is rotated at its upper end and the run-out is measured at the lower end (TIR), then $TIR = 2L\theta$ and

$$\frac{\sigma_b}{\sigma_T} = \frac{4}{d}\,(TIR)$$

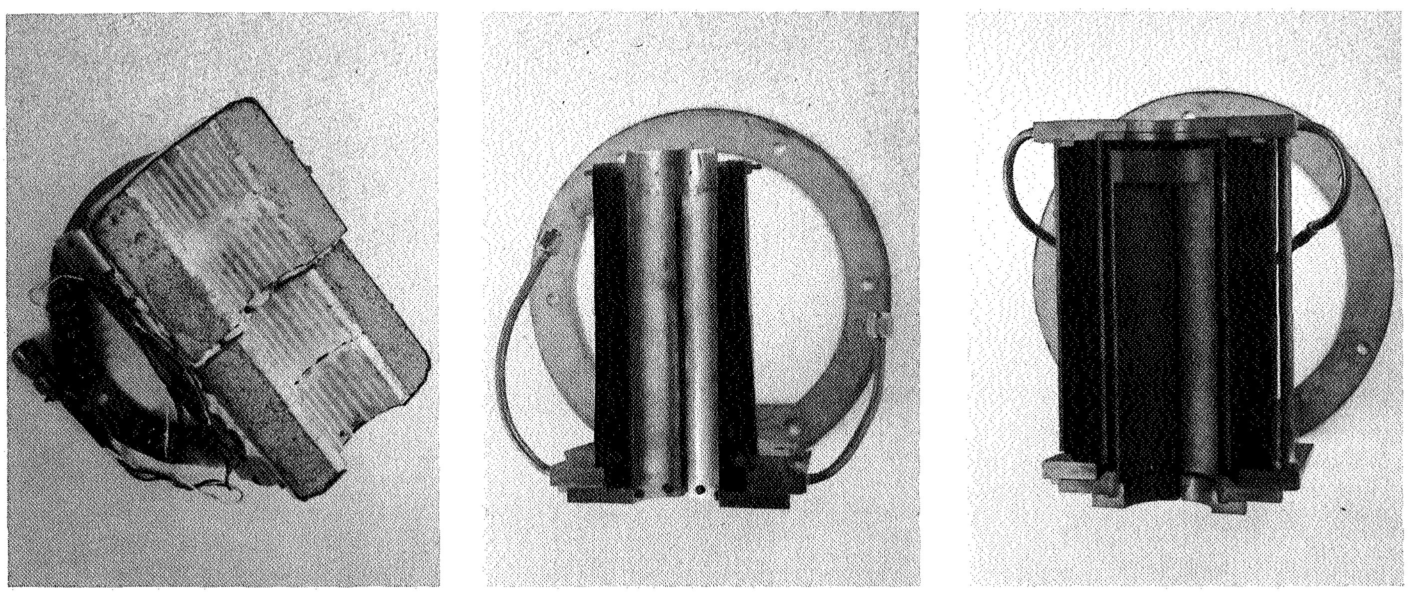
For this configuration, $d = 0.15$ and $\sigma_b/\sigma_T = 2.7\%$ for 0.001 TIR.

V. Environmental System

The primary function of the environmental system in this facility was to provide high temperatures. Atmospheric effects have been reported in oxides. However, the evidence is still uncertain, and such an effect can be expected to be less controlling than such factors as grain size or temperature. In the case of carbides at high temperatures, protection from oxidation is required.

The environmental system used consists of a front opening water-cooled chamber having capabilities for moderate vacuum or sealed gas atmosphere. Three interchangeable furnace units may be placed in the chamber:

(1) A conventional nickel-chrome alloy furnace may be used to 1200°C in oxidizing or neutral environments. Temperatures are sensed by a platinum



1200°C MAXIMUM IN OXIDIZING OR NEUTRAL ATM

1700°C MAXIMUM IN NON-OXIDIZING ATM

2500°C MAXIMUM IN NON-OXIDIZING ATM

Fig. 10. Environmental furnaces

thermistor element which controls a 2-kw SCR power supply. Commercially available heating elements are used and are connected so as to load a 220-V line.

(2) A water-cooled molybdenum sheet furnace, using backup molybdenum radiation shields, may be used to 1700°C in neutral or reducing atmospheres. Temperatures may be sensed by a thermocouple and used to control a 50-kV-A 30-V power supply. The molybdenum elements could be replaced by tantalum or tungsten, should higher temperature operation of this type of furnace be required.

(3) A water-cooled graphite furnace, using a combination of graphite radiation shields and graphite felt insulation, is available for use to 2500°C in a neutral atmosphere. This furnace is primarily for use with the carbides. Temperature sensing can be made by means of a total radiation parameter controlling the same power suppliers as used with the molybdenum furnace. However, control is generally maintained manually.

The various furnaces are shown in Fig. 10. All units employed the *clam-shell* design with half of the heating element and shielding mounted within the environmental chamber and the other half mounted on the door.

VI. Evaluation of System

To evaluate the overall capability of the system to provide accurate mechanical data, the series of results shown in Table 2 were generated on well-known engineering materials as shown on the sample stress-strain curve in Fig. 11. The data in Table 2 indicate that alignment is important even when measurements are made on ductile materials such as these.

VII. Conclusions

A facility has been designed and constructed for the testing of brittle materials in tension and will provide accurate data within known limits. The mechanical behavior of a specimen may be determined with the following limits.

(1) Average stress $\leq 1\%$ (load/unit area).

(2) Local stress $\leq 5\%$.

(3) Strain sensitivity approximately 2×10^{-5}.

(4) Strain precision $2\% + 2 \times 10^{-5}$.

(5) Meaningful modulii with strains of 2.5×10^{-3}.

Table 2. Modulus of elasticity of standard materials[a]

Material	Gage length, mm	Contour	Stress range, kg/mm²	Modulus of elasticity, kg/mm² × 10⁴	Remarks
4130 steel	38.1	No	0–8.8	2.14	
4130 steel	38.1	No	8.8–35.1	2.05	
4130 steel	38.1	No	35.1–57.6	2.10	
1113 steel	19.0	Yes	7.0–17.6	1.87	Specimen misaligned
1113 steel	19.0	Yes	7.0–17.6	2.03	
1113 steel	12.7	Yes	0–17.6	2.23	
1113 steel	12.7	Yes	0–17.6	2.23	
6061-T6 Al	38.1	No	0–3.5	0.78	
6061-T6 Al	38.1	No	3.5–17.6	0.67	
6061-T6 Al	38.1	No	17.6–23.7	0.63	
6061-T6 Al	38.1	No	1.7–8.0	0.65	Specimen misaligned

[a]Standard values for steel and aluminum are 2.05×10^4 kg/mm² and 0.75 kg/mm², respectively.

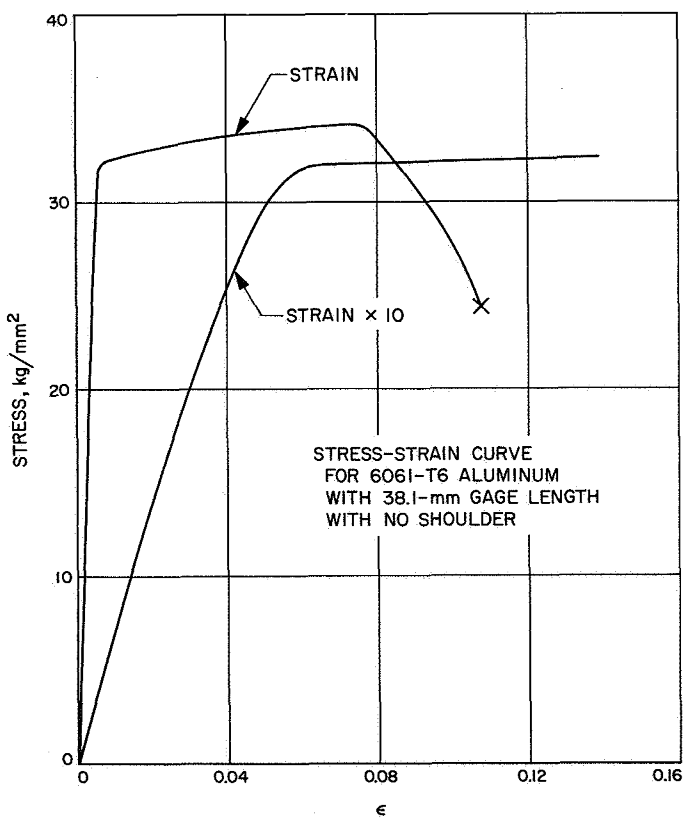


Fig. 11. Stress-strain curve for 6061-T6 aluminum

Appendix

Inspection Procedure Alumina or TaC Tensile Specimens

1. Visually inspect parts for surface defects, chipped edges, chatter marks, etc.

2. Mount specimen on one end, vertical to surface plate, and measure parallelism of ends with tenth indicator. If reading is more than 0.0002 *TIR*, part is not acceptable and should be rejected to vendor for rework.

3. Mark ends at high and low points with pencil.

4. Mount specimen on precision rotary table, vertical to reference base, and center to zero read-out. Check concentricity of reduced area of specimen and opposite end. Deviations, if any, greater than tolerance are cause for rejection.

5. Optically check perpendicularity of surfaces *B* and *C*. Any angularity to 0.400 and 0.500 diam greater than tolerance call-out will be cause for rejection.

6. Check contour, if any, using contour mask. Discontinuity in contour surface or deviation greater than tolerance call-out for straightness of 0.150 diam section will be cause for rejection.

7. Make photographic reproduction of specimen, approximately 8×10 in. size, that will show match of specimen to contour mask. Take two shots per specimen, i.e., one parallel to pencil marks on end and one perpendicular. Prints to be marked with specimen number and position location. Photo should also include partial view of 0.400 and 0.500 diam.

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